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Manuscript title: On the development of an Ultra-High Capacity Tensiometer capable of measuring water tensions to 7 MPa

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ABSTRACT

Tensiometers are increasingly used in geotechnical engineering to monitor pore-water tension in the field and to study the hydro-mechanical behaviour of unsaturated soils in the laboratory. Early tensiometers exhibited a relatively small measuring range, typically limited to a tension of 0.1 MPa, due to water tension breakdown inside the sensing unit at absolute negative pressures. This limitation was subsequently overcome by the design of High Capacity Tensiometers (HCT), which enabled the measurement of considerably larger pore-water tensions. According to literature, the highest value of water tension ever recorded by a HCT is equal to 2.6 MPa. In the present work, this value is almost tripled by designing a novel Ultra-High Capacity Tensiometer (UHCT) capable of recording water tensions up to 7.3 MPa. This is achieved by replacing the traditional ceramic interface with a nanoporous glass (typically employed by physicists for the study of confined liquids), which has never been used before for the manufacture of tensiometers. The maximum attainable tension has been determined via tests where the UHCT measurement was progressively increased by vaporising water from the glass interface until the occurrence of tension breakdown (often referred to as “heterogeneous cavitation” or “tensiometer cavitation”). The increased measuring range and the potentially larger measuring stability of the proposed UHCT will contribute to enhance laboratory testing of soils at high suctions and long-term monitoring of earth structures.

KEYWORDS: Water tension measurement, tensiometer, capillary pressure, suction, unsaturated soils.

Introduction

The tensile strength of water has been widely studied by physicists using equipment like miniature Berthelot tubes (Berthelot, 1850). For example, Zheng et al. (1991) measured a tensile stress of 140 MPa in a single water crystal, a value believed to be close to the strength of water.

Soil pore-water tension (often referred to as “suction”) is a key variable in several geotechnical applications including slope stability, foundation settlements, excavations, and retaining structures. In all these applications, pore-water tension can attain values well beyond 0.1 MPa and can therefore only be measured by sensors capable of recording absolute negative pressures.

The first high capacity tensiometer (HCT) measuring pore-water tensions up to 1.5 MPa was developed at Imperial College London by Ridley and Burland (1993). A schematic view of this sensor is shown in Figure 1 where it is possible to distinguish three main components: a pressure transducer, a water reservoir and a porous ceramic interface. Since then, a number of similar designs have been proposed in the literature (Guan and Fredlund, 1997; Meilani et al., 2002; Tarantino and Mongiovì, 2002; Take and Bolton, 2003; Lourenço et al., 2008; Cui et al., 2008; Mendes and Buzzi, 2013; Mendes et al., 2016).

When the porous interface of a saturated HCT is placed in contact with a soil at a given suction, water is drawn out of the reservoir and the recorded tension increases until equilibrium with the external suction is attained. If the recorded water tension exceeds a value of 0.1 MPa, and therefore enters the range of negative absolute pressures, the water inside the reservoir is in a metastable state (Marinho et al., 2008) and tension breakdown will occur at some point, the sooner the higher the tension value. This breakdown, which is often

termed “heterogeneous cavitation” or “tensiometer cavitation”, consists in the expansion of a small air nucleus, which grows to occupy the entire reservoir volume.

Moreover, suction can only be measured if capillary menisci form at the outer boundary of the ceramic interface to sustain the pressure differential between external atmospheric air and internal tensile water. These menisci prevent air from breaking inside the sensor, thus allowing it to remain saturated. The maximum sustainable pressure differential is named “air entry value” (AEV) and is inversely related to the size of the largest pore in the interface as demonstrated by Mendes et al. (2016). Thus, the AEV of the interface constitutes a limit to the maximum tension that can be measured by HCTs.

The above discussion indicates that the two main challenges in the design of HCTs are: i) to prevent occurrence of heterogeneous cavitation inside the sensing unit and ii) to increase the AEV of the porous interface. By doing so, it is possible to extend both the maximum measurable tension and the stability of measurements over time.

Interestingly, Mendes and Buzzi (2013) demonstrated that heterogeneous cavitation is initiated inside the voids of the porous interface, although it eventually manifests itself in the water reservoir. Similar conclusions were drawn by Tarantino and Mongiovì (2001). This evidence together with the lesson learned by water physicists would suggest that heterogeneous cavitation could be countered by decreasing the pore size of the interface. A finer porous interface could therefore simultaneously increase the AEV and reduce the likelihood of heterogeneous cavitation inside its voids.

Consistent with the above hypothesis, this paper presents the study of a novel Ultra-High Capacity Tensiometer (UHCT) that incorporates a porous interface with extremely small pores to extend the range and stability of suction measurements.

Ultra-High Capacity Tensiometer (UHCT)

The Ultra-High Capacity Tensiometer (UHCT) presented in this paper incorporates a novel glass interface, whose largest pore measures only a few nanometres compared to the hundreds of nanometres of standard ceramics. This nanoporous glass is typically used by physicists for studying the interaction between solid and liquid phases under confined conditions. The glass interface is chemically inert, like conventional ceramics, but is also more fragile than conventional ceramics. Apart from the finer porous glass interface and a pressure transducer with a larger measuring range of 35 MPa to record higher tensions, the proposed UHCT is similar to the HCT of Mendes and Buzzi (2014) and is shown schematically in Figure 2.

Figure 3 shows two photographs of the proposed UHCT without the glass interface (Figure 3a) and with the glass interface (Figure 3b), respectively. Figure 3b also shows that, when saturated, the glass interface becomes transparent, thus allowing visual inspection of the water reservoir and pressure transducer.

Table 1 compares the main properties of the glass interface used in this study with those of a typical ceramic interface with a nominal AEV of 1.5 MPa, as per manufacturer specifications. Figure 4 shows instead the pore size distribution of the two materials measured by mercury intrusion and nitrogen adsorption porosimetry. Each test is labelled according to the format x_y, where x refers to the type of porous interface (ceramic or glass) and y refers to the type of test (MIP for mercury intrusion or NA for nitrogen adsorption). Inspection of Figure 4 indicates that the largest ceramic pore has a diameter between 150 nm and 200 nm (Figure 4a) and is therefore considerably bigger than the largest glass pore, which has a diameter of only 7 nm (Figure 4b).

Under the simplifying hypotheses of cylindrical pores and zero contact angle, the AEV is inversely related to the largest pore diameter, d according to the Young-Laplace equation:

$$AEV = \frac{4\gamma}{d} \quad [1]$$

where γ is the air-water surface tension (72.8 mN/m at 20 °C). According to Equation (1), the nominal AEV of 1.5 MPa, for the ceramic tested in the present work, would correspond to a diameter of about 190 nm, a value that matches well the size of the largest pore measured from MIP tests (Figure 4a). Instead, a diameter of 7 nm for the largest pore of the glass interface (Figure 4b) would correspond to an AEV of about 42 MPa according to Equation (1).

Figure 4 also shows the pore diameters calculated by Equation (1) for an AEV of 0.5 MPa, which is the lowest AEV of standard ceramics employed during manufacture of HCTs, and 7 MPa, which corresponds approximately to the maximum tension measured in the present work.

Saturation and calibration

The UHCT was saturated with de-aired water according to the procedure described by Mendes and Buzzi (2014). During the first saturation from dry conditions, the porous interface was exposed to high vacuum ($\cong 10^{-10}$ MPa, absolute pressure) for at least 2 hours before being pressurised with compressed water at 15 MPa for 48 hours. This pressure is considerably higher than that applied during saturation of conventional HCTs, which usually does not to exceed 3-4 MPa, and is justified by the significantly smaller pores of the glass interface compared to standard ceramics.

The UHCT was then calibrated in the positive pressure range by imposing a compression-decompression cycle between 0.05 MPa and 15 MPa. During this cycle, water pressure was changed in steps of variable sizes with smaller increments in the low pressure range. The

results of the calibration cycle are shown in Figure 5 (black line), where the relationship between pressure and voltage appears proportional. The linear calibration equation of Figure 5 was therefore defined over the positive pressure (compression) range and then extrapolated to the negative pressure (tension) range, as suggested by Tarantino and Mongiovì (2002) and confirmed by Lourenço et al (2008).

After occurrence of tension breakdown during tests, the UHCT was re-saturated to restore its ability to measure tension by applying a water pressure of 15 MPa to the ceramic interface for a period between 12 and 72 hours. Unlike the first saturation, no vacuum was applied in this case because the sensor was already filled by water with the presence of only small air cavities that expand upon tension breakdown.

Figure 5 also shows another calibration equation (grey line), which was determined after repeated water tension breakdowns. Like for conventional HCTs, water tension breakdowns produce a drift with constant slope of the calibration equation. To correct this drift, the UHCT was therefore placed in free water and the calibration was re-zeroed before each test.

Results and discussion

The maximum tension sustained by the UHCT was determined by exposing the glass interface to the atmosphere so as to vaporize water and measure progressively larger tensions until the occurrence of tension breakdown. Figure 6 shows the results from three initial evaporation tests performed after re-saturation periods of only 12 hours. Inspection of Figure 6 indicates that the maximum measured tension varied between 2.5 MPa and 2.9 MPa, which was a rather disappointing result given that the AEV of the glass was much higher and equal to 42 MPa according to Equation (1). A possible reason of this behaviour was the occurrence of early heterogeneous cavitation due to poor saturation of the glass interface caused by pressurisation at only 15 MPa. A pressure of 15 MPa is, in fact, considerably lower than the

AEV of the glass and hence insufficient to dissolve all entrapped air. To facilitate elimination of any residual air, the pressurisation time was increased from 12 hours to 72 hours. An increase of pressure above 15 MPa was instead ruled out because of potential damages to the fittings of the saturation system.

The results from the subsequent evaporation tests are shown in Figure 7, which indicates that a longer pressurisation time increased the maximum sustainable water tension to 4.3 MPa, 5.0 MPa, 6.0 MPa and 7.1 MPa after the first, second, third and fourth re-saturations, respectively. After the third and fourth re-saturation, the variation of recorded tension with time became markedly irregular with sudden jumps of the readings. This was attributed to the formation of a crack inside the nanoporous glass parallel to the sensing face, as also confirmed by visual inspection. The influence of this crack on the UHCT response was only evident after the third re-saturation but invisible micro-cracks had probably started to form earlier.

At this stage, it is also not possible to know whether cracking of the glass interface occurs during saturation, due to compressive pressurisation, or during evaporation tests, due to water tension breakdown. The latter hypothesis seems however more realistic because of the instantaneous release of water tension, which generates a sudden mechanical shock on the glass and favours a brittle response that promotes fracture. Instead, the relatively slow increase of compressive stress during pressurization tends to favour a ductile response and hence delays fracture.

Despite the formation of a crack inside the porous interface, the UHCT was still capable of measuring high tensions because of the presence of an intact glass layer separating the external atmosphere from the inner water reservoir. Yet, from the fourth re-saturation onwards, the measuring range did not increase further and stabilized at around 7 MPa. This is shown in Figure 8, where the results from seven subsequent evaporation tests are reported. It

was during one of these later tests that the highest water tension of 7.3 MPa was measured. This value is about three times higher than the water tension of 2.6 MPa recorded by Tarantino and Mongiovi (2001), which represents the maximum tension ever recorded by a HCT.

Figure 8 also shows that evaporation curves were no longer smooth, as during earlier tests, but tended to exhibit a multi-modal shape with two pressure plateaus, one at a relatively low tension of 0.1 MPa and another one between 1.5 MPa and 3.5 MPa. The earlier plateau at 0.1 MPa is likely caused by the presence of a large crack parallel to the glass interface, which impedes the increase in water tension until the void is completely de-saturated. The later plateau, between 1.5 MPa and 3.5 MPa, is likely caused by the desaturation of smaller cracks inside the remaining glass layer that seals the water reservoir.

A second UHCT prototype was built to corroborate the above findings. In this case, the pressurisation time was fixed at 72 hours from the beginning. Figure 9 shows the results obtained with this second prototype, which consistently recorded maximum water tensions between 5.1 MPa and 6.5 MPa. As with the first prototype, a crack in the glass started to form parallel to the sensing face after some tests, which limited the further increase of the maximum tension.

Inspection of all evaporation curves indicates that the rate at which tension increases with time does not slow before breakdown. Typically, in conventional HCTs, a progressively slower rate is observed as the recorded tension approaches the AEV of the porous interface (Mendes and Buzzi, 2013). The approximately constant rate observed in the present work suggests, therefore, that the AEV of the porous glass is significantly larger than the maximum recorded tension and the observed breakdown is caused by heterogeneous cavitation rather than air breakthrough. This is also consistent with the theoretical AEV of 42 MPa of the glass

interface as predicted by Equation (1) from the pore size distribution of Figure 4b. Much of the measuring potential of the UHCT remains therefore untapped due to the fragility of the glass interface that cracks when subjected to the mechanical shock of tension breakdown. The formation of a crack then facilitates the occurrence of heterogeneous cavitation, which in turn impedes the achievement of larger tensions. Overcoming this technical limitation might further extend the sensor range, possibly up to a water tension of 42 MPa.

The above results confirm that a finer interface not only increases the AEV but also delays the occurrence of heterogeneous cavitation compared to standard HCTs and therefore extends the measuring range of the sensor. A finer interface may also enhance the stability of measurements over time as heterogeneous cavitation is the prime cause of tension breakdown in metastable water subjected to prolonged negative absolute pressures.

The preliminary results presented in this paper explore the effect of a nanoporous interface on the standard evaporation curve that defines the measuring range of tensiometers. However, the current version of the sensor cannot measure suction changes in soils as cracking of the glass interface precludes hydraulic continuity between the soil and the sensor. The authors are presently working on alternative nanoporous interfaces that do not crack and can therefore allow continuous measurements of soil suction.

Conclusions

This manuscript has presented a novel Ultra-High Capacity Tensiometer (UHCT) capable of measuring water tensions in excess of 7 MPa. This is about a threefold increase with respect to the maximum value of water tension ever reported in the literature for conventional High Capacity Tensiometers (HCT). At the core of the proposed UHCT there is a novel glass interface with pore sizes of the order of nanometers compared to the hundreds of nanometers of standard high air entry value ceramic interfaces.

The maximum sustainable tension of the proposed UHCT has been determined by means of evaporation tests where water tension is increased by exposing the nanoporous glass interface to the atmosphere until tension breakdown occurs due to heterogeneous cavitation. The maximum measured water tension increases with consecutive tension breakdowns, due to the progressive elimination of entrapped air nuclei, until attaining a measurement limit of about 7 MPa. This measuring limit corresponds to the appearance of a crack inside the nanoporous glass along a plane parallel to the sensing face. This crack is probably caused by the mechanical shock associated with the sudden release of stress upon tension breakdown. It is expected that, if fracturing of the nanoporous interface could be prevented, the maximum sustainable tension could be further increased, possibly to 42 MPa which is the estimated AEV of the glass.

The present study has focused on the development of a novel UHCT and on the definition of its measuring limit. The current UHCT cannot measure suction changes in soils due to the formation of a sub-horizontal crack in the glass interface that impedes hydraulic continuity between the soil and the sensor. Future research will therefore concentrate on the adaptation of the proposed UHCT for measuring suction in soils as well as on the extension of its measuring range.

Acknowledgments

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Table 1 - Properties of porous interfaces.

Porous interface	Ceramic	Glass
Chemical composition	Kaolinitic clays	96% SiO ₂ , 3.6% B ₂ O ₃ and 0.4% Na ₂ O
Porosity	32%	28%
Bulk density	1.5g/cm ³	
Largest pore size (diameter)	100-200 nm	7 nm

Figure 1. Imperial College High Capacity Tensiometer (Ridley and Burland, 1993).

Figure 2. Proposed Ultra-High Capacity Tensiometer.

Figure 3. Ultra-High Capacity Tensiometer without nanoporous glass interface (a) and with a saturated nanoporous glass interface (b).

Figure 4. Pore size distribution of interfaces: a) ceramic and b) glass.

Figure 5. Initial calibration curve (black) and drifted calibration curve (grey).

Figure 6. Evaporation tests after 12 hours re-saturation.

Figure 7. Evaporation tests after 72 hours re-saturation.

Figure 8. Evaporation tests after localized cracking of the glass interface.

Figure 9. Evaporation tests performed with the second Ultra-High Capacity Tensiometer prototype.

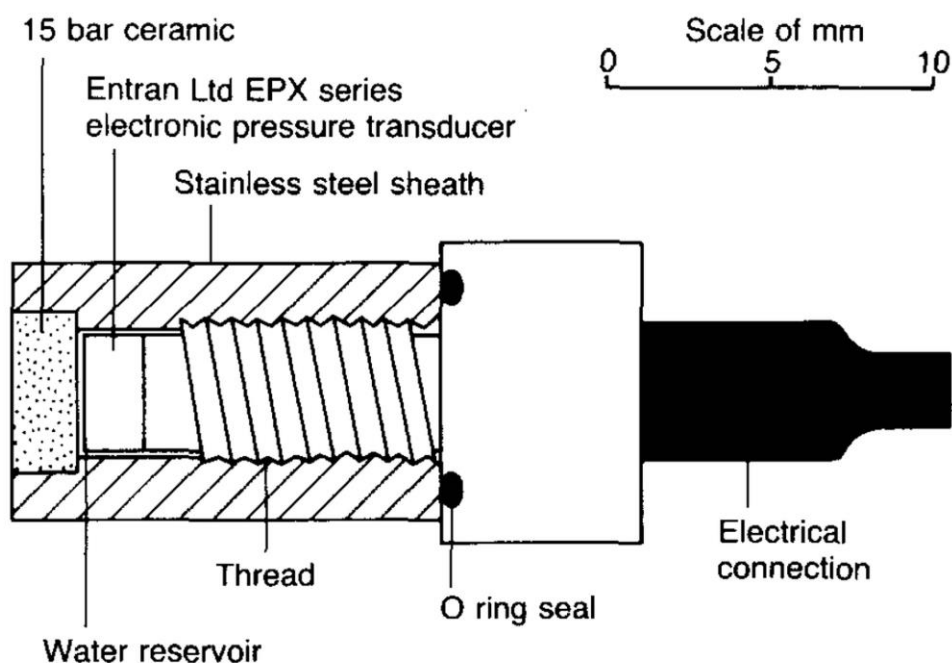


Figure 1

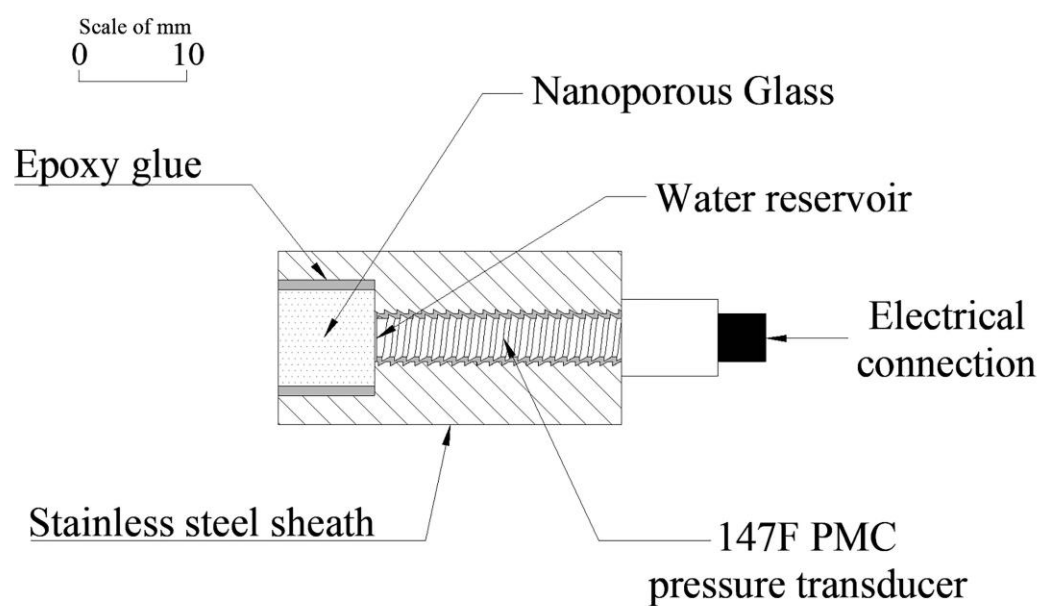
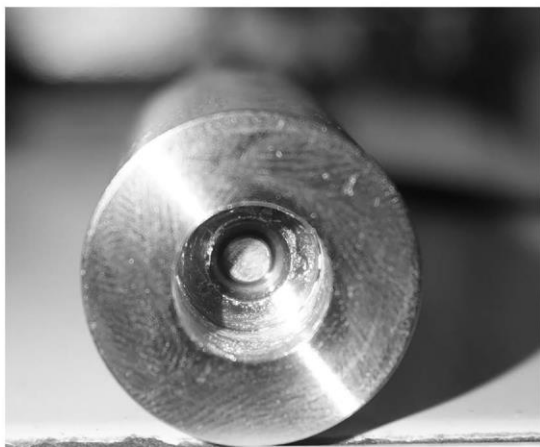


Figure 2



a)

Figure 3a

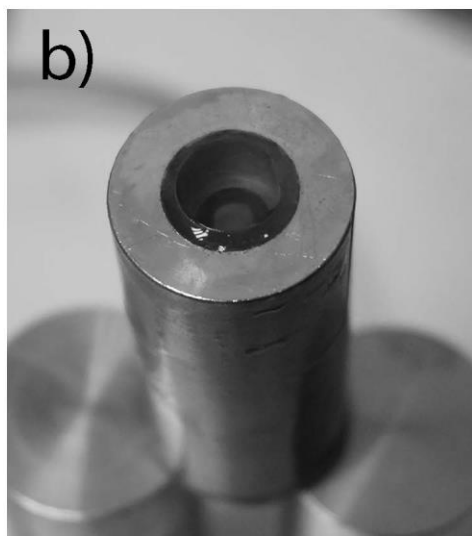


Figure 3b

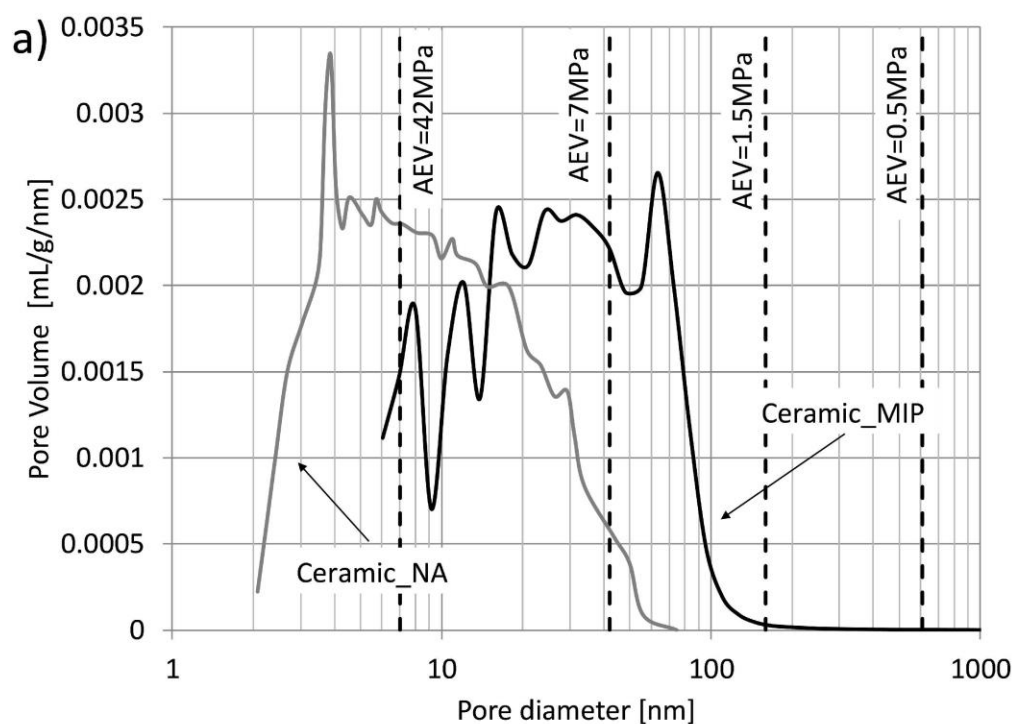


Figure 4a

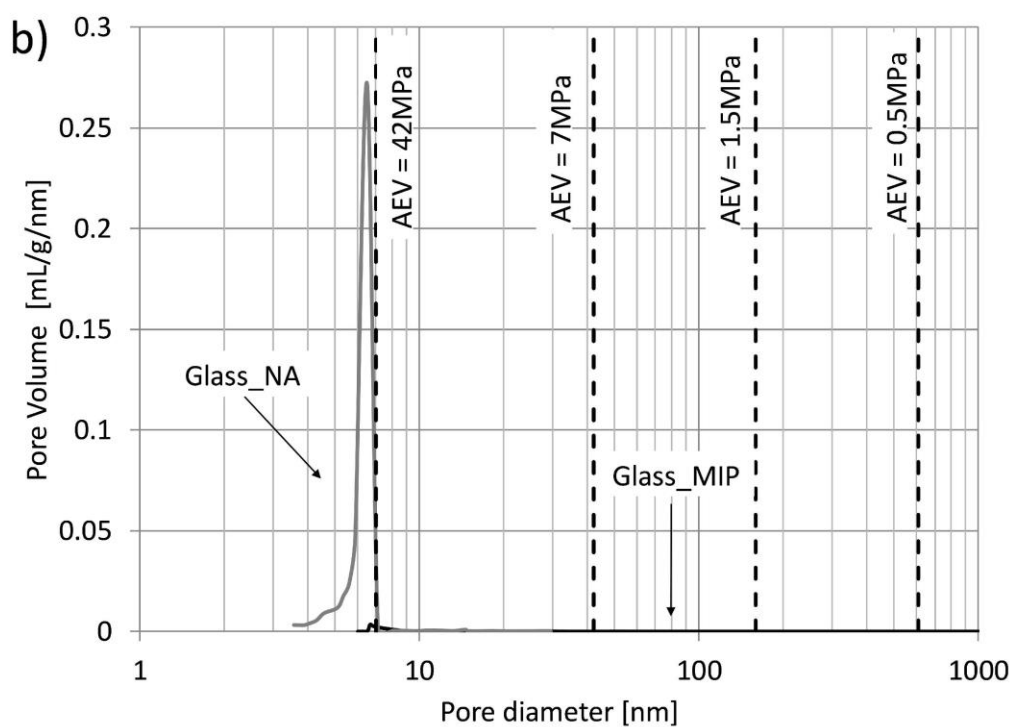


Figure 4b

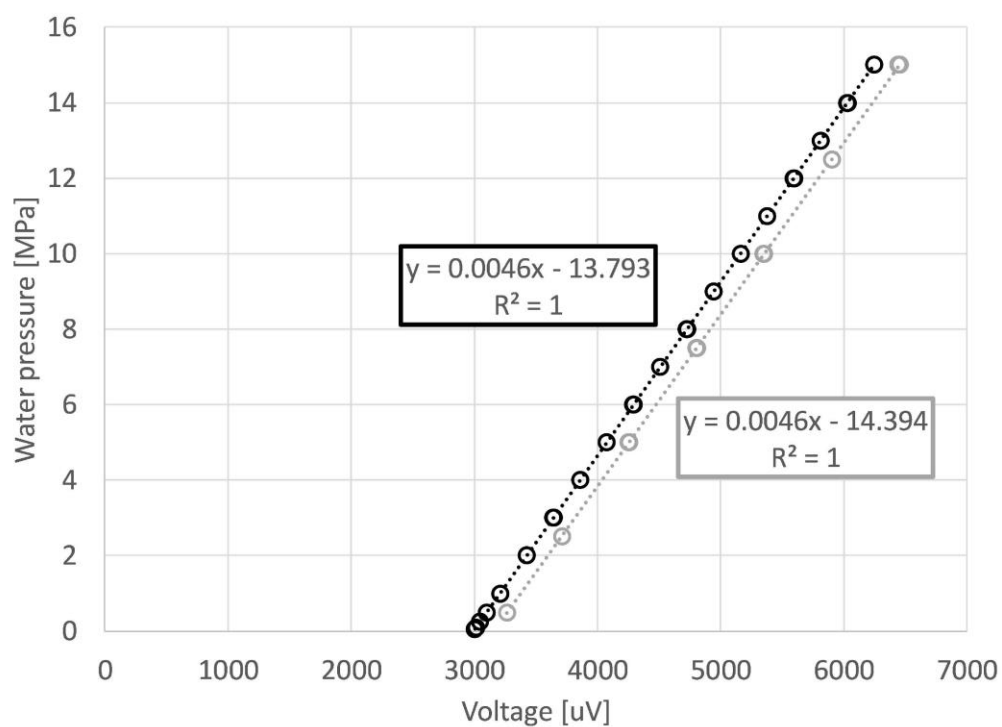


Figure 5

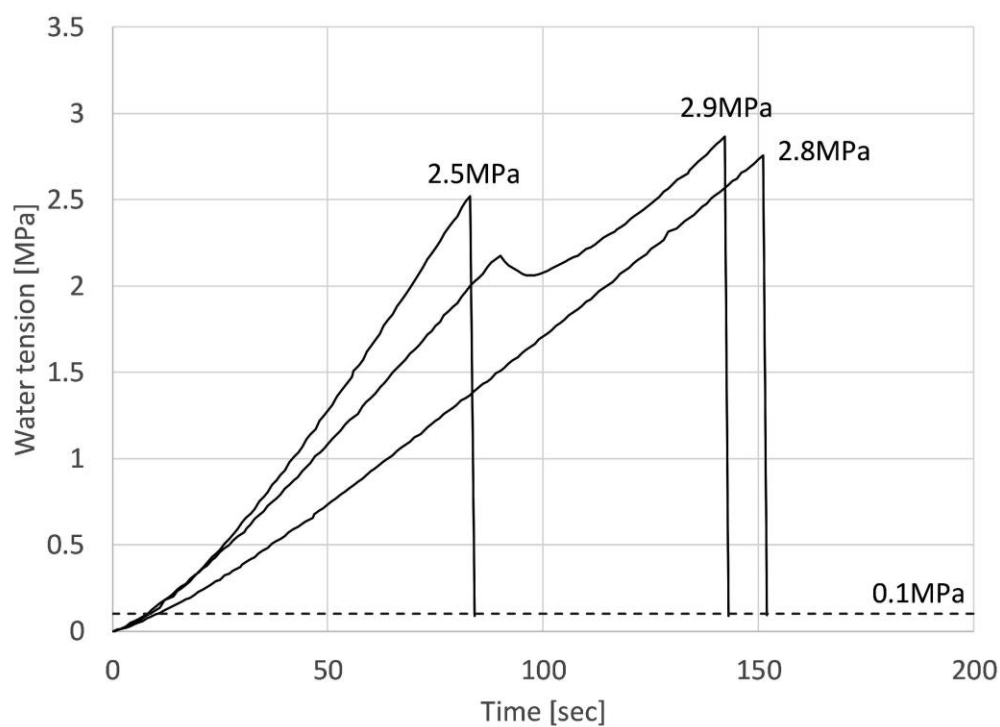


Figure 6

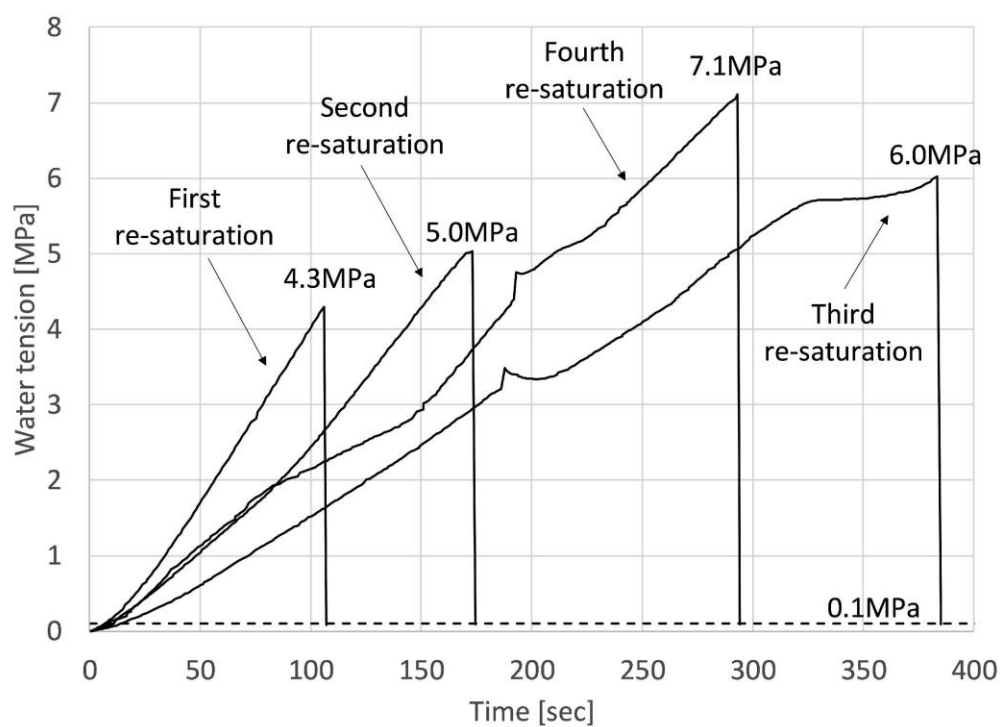


Figure 7

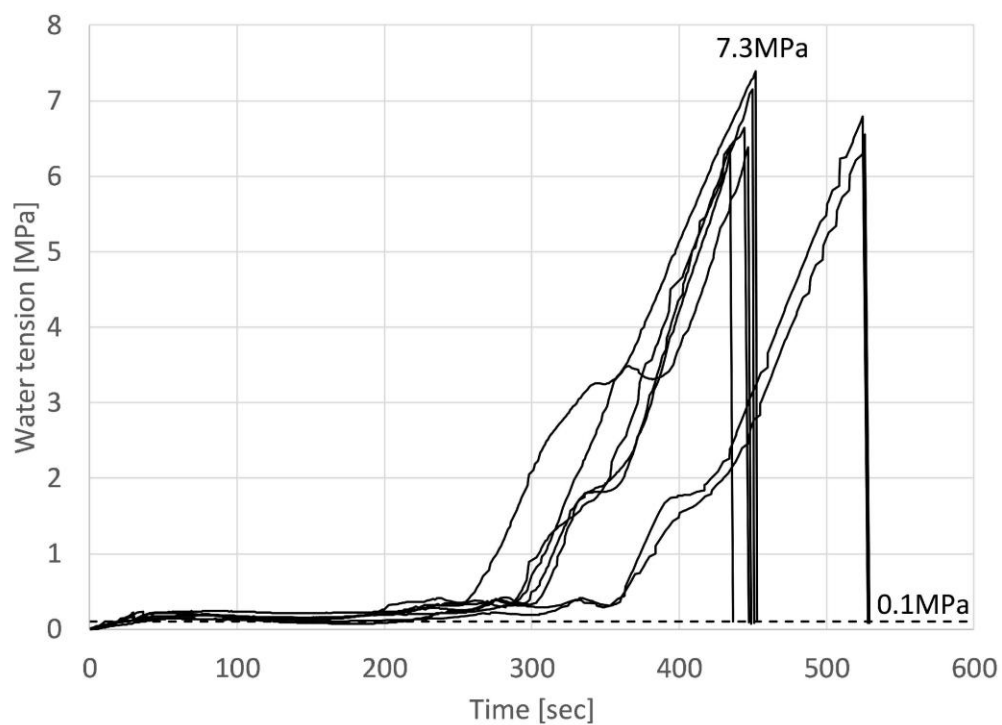


Figure 8

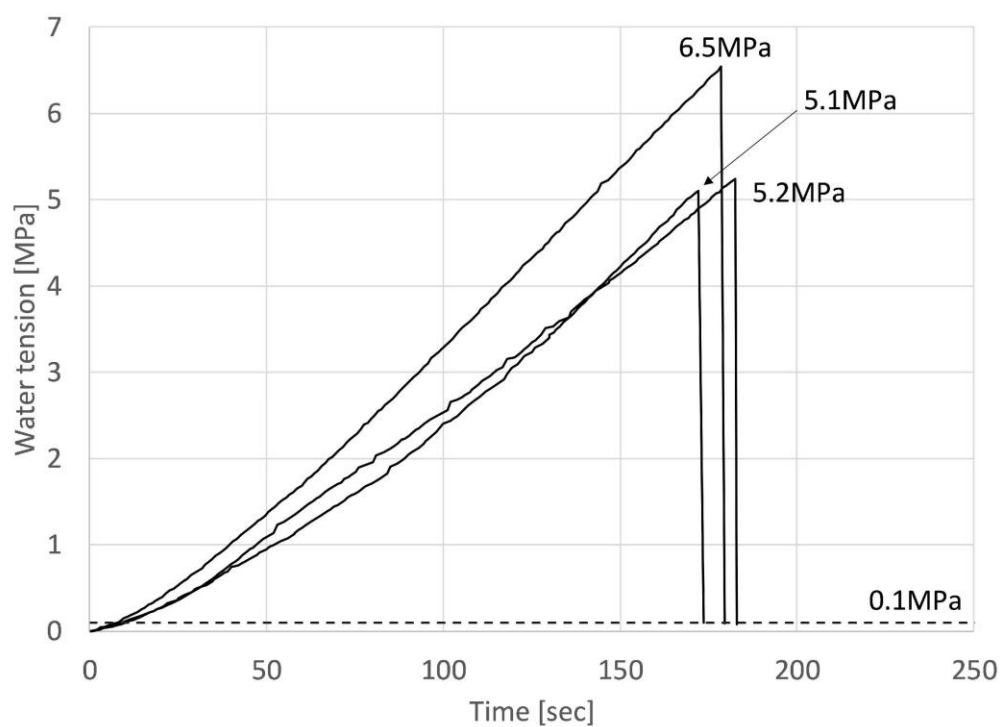


Figure 9